

STAT

COLORIMETRIC DETERMINATION OF LITHIUM

authors: A. V. Nikolayev, A. A. Sorokina

Source:

DAN SSSR, Vol LXXVII, No. 3, pp 427-428

STAT

STAT

COLORIMETRIC DETERMINATION OF LITHIUM

A. V. Nikolayev, A. A. Sorokina,

Moscow Inst of Nonferrous Metals and Gold imeni

M. I. Kalinin

V. I. Kuznetsov /1/, who proposed a qualitative color reaction for lithium, pointed out that it ^{also} must be possible to carry out a quantitative colorimetric determination, and of lithium, and mentioned that nobody had ever done this before.

Benzene-2-arsonic acid - (1-azo-1)-2-hydroxynaphthalene-3,6-disulfonic acid (Kuznetsov's reagent) is very sensitive, so that it should be used only on small quantities of lithium. Colorimetric determinations were carried out by visual comparison with a scale ranging from 0.175 to 0.50 mg of Li with intervals of 0.025 mg of Li between individual stages. The solution being tested and the standard solutions were brought to a volume of 50 ml.

In cases when the determination is done on a pure lithium ~~the~~ salt, 2.5 ml of a 20% KOH solution and 0.5 ml of a sodium stearate solution are added after the solution has been diluted to 50 ml. At the expiration of 15 min, 0.9 ml of a solution of Kuznetsov's reagent (0.1 g in 100 ml of water) are added into the test tubes and a comparison of the sample being analyzed with the standard scale is made.

It is advisable to carry out the determination twice or take the average of determinations carried out on two aliquot portions.

In view of the fact that salts of Na, K, Mg, and Ca always accompany Li, and are often present in predominant quantities, it was important to investigate their influence on the quantitative determination of Li. It was established that sulfates and chlorides of K and Na do not affect the color reaction when their ratio with reference to Li is 250:1, 300:1, or 500:1. Ca and Mg do interfere with the determination of Li: 5-6% of Ca and Mg (with reference to Li) produce no effect, but 14-17% of Ca ^{and} Mg lead to values which are too high. The necessity of ^{separating out} determining Ca and Mg before the determination of Li therefore arose. In doing this, the best results were achieved by using K₂CO₃ (20%

CONFIDENTIAL

RESTRICTED

of /7/ solution). One must avoid an excess of K_2CO_3 : a 0.1 g excess in 50 ml noticeably lowers the intensity of coloration produced by Li, which leads to values that are too low (cf. Table 1).

In order to check the accuracy of the method in the presence of K, Ca, and Mg, solutions containing 12.5-25 mg of Li, 4-5 mg of CaO , 1-6 mg of MgO , and 0.5 g of K_2SO_4 were made up. Ca and Mg were then precipitated with K_2CO_3 . After the precipitate had been filtered off, KOH and Kuznetsov's reagent were added, whereupon the colorimetric determination was carried out (the end volume was 50 ml). ^{A number of} ~~The results~~ filtrate samples was neutralized with HCl in order to eliminate the effects of an excess ~~also listed in Table 2~~ of K_2CO_3 . The results are listed in Table 2.

Separation of Ca and Mg with ammonium carbonate was also attempted. The filtrate obtained after this precipitation was evaporated to dryness and the ammonium salts were then driven off by heating.

Furthermore, a special determination of Li by our method in the precipitate obtained according to Smith-Gooch's procedure was conducted. It turned out that the method proposed by us is more accurate. The results obtained on this are given in Table 3.

An attempt to use a photoelectric colorimeter for determining lithium was unsuccessful.

Checking of the method under plant laboratory conditions indicated closer agreement of the results of parallel tests, gain in time, simplification of the analytical procedure, saving in labor, and advantages gained by eliminating ^{the use of} ~~as well as~~ alcohol ~~and~~ reducing the demand for platinum dishes. The method proposed increases the efficiency of production control, thereby expediting production.

The drawbacks of the method are the difficulty of distinguishing between shades and the necessity of using daylight or a daylight lamp.

Bibliography.

1. V. I. Kuznetsov, Zhur. Anal. Khim., Vol. III, No 5, p. 295 (1948).

RESTRICTED

RESTRICTED

of /?/ solution). One must avoid an excess of K_2CO_3 : a 0.1 g excess in 50 ml noticeably lowers the intensity of coloration produced by Li, which leads to values that are too low (cf. Table 1).

In order to check the accuracy of the method in the presence of K, Ca, and Mg, solutions containing 12.5-25 mg of Li, 4-5 mg of CaO , 1-6 mg of MgO , and 0.5 g of K_2SO_4 were made up. Ca and Mg were then precipitated with K_2CO_3 . After the precipitate had been filtered off, KOH and Kuznetsov's reagent were added, whereupon the colorimetric determination was carried out (the end volume was 50 ml). ^{A number of} the results of the filtrate samples was neutralized with HCl in order to eliminate the effects of an excess of K_2CO_3 . The results are listed in Table 2. ~~also listed in Table 2~~

Separation of Ca and Mg with ammonium carbonate was also attempted. The filtrate obtained after this precipitation was evaporated to dryness and the ammonium salts were then driven off by heating.

Furthermore, a special determination of Li by our method in the precipitate obtained according to Smith-Gooch's procedure was conducted. It turned out that the method proposed by us is more accurate. The results obtained on this are given in Table 3.

An attempt to use a photoelectric colorimeter for determining lithium was unsuccessful.

Checking of the method under plant laboratory conditions indicated closer agreement of the results of parallel tests, gain in time, simplification of the analytical procedure, saving in labor, and advantages gained by eliminating ^{the use of} alcohol ~~and~~ as well as reducing the demand for platinum dishes. The method proposed increases the efficiency of production control, thereby expediting production.

The drawbacks of the method are the difficulty of distinguishing between shades and the necessity of using daylight or a daylight lamp.

Bibliography.

1. V. I. Kuznetsov, Zhur. Anal. Khim., Vol. III, No 5, p. 295 (1948).

RESTRICTED

Table 1.

Mgs of Li taken	Content of K_2CO_3 in g per 50 ml	Mgs of Li found	Error in %
0.250	0.10	0.248	- 0.8
0.275	0.10	0.260	- 5.5
0.400	0.10	0.387	- 3.2
0.250	0.06	0.250	0.0
0.275	0.06	0.275	0.0
0.400	0.06	0.400	0.0

Table 2.

Mgs of Li taken	Mgs of CaO taken	Mgs of MgO taken	Grams of K_2SO_4 taken	Mgs of Li found with neutralization	Mgs of Li found without neutralization	Excess of K_2CO_3 in mgs
12.5	4	1	0.5	12.5	12.5	20
12.5	5	6	0.5	12.5	12.5	20
25.0	4	1	0.5	24.0	24.0	50
25.0	5	6	0.5	24.8	24.0	50

RESTRICTED

Taken for analysis.				Table 3.							
Li in mg	CaO in mg	MgO in mg	K_2SO_4 in g	Mgs of Li found colorimetrically (precipitation method) with K_2CO_3	Error in %	Mgs of Li found colorimetrically (precipitation with ammonium carbonate)	Error in %	Mgs of Li found gravimetrically	Error in %	Check of the gravimetric method by colorimetric determination in precipitate	Error in %
25 12.5	5 5	6 6	0.5 0.5	25 12.5	0.0 0.0	24.8 13.0	- 0.8 + 4.0	26 15.8	+ 4.0 + 26	25.2 12.0	+ 1.5 - 4.0
											5.

CONFIDENTIAL